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IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF	:
TOSHIO MORII, ET AL.	: EXAMINER: MARCHESCHI, M.A.
SERIAL NO: 10/718,573	: GROUP ART UNIT: 1755
FILED: NOVEMBER 24, 2003	: RCE FILED: SEPTEMBER 8, 2005
FOR: HIGH CONCENTRATION SILICA SLURRY	:

STATEMENT REGARDING PRIORITY DOCUMENT AND

ENGLISH-LANGUAGE TRANSLATION OF PRIORITY DOCUMENT

COMMISSIONER FOR PATENTS
ALEXANDRIA, VIRGINIA 22313

SIR:

Applicants note that the attached certified copy of the JP 2002-339402 priority document and the attached English-language translation of the priority document differ from the photocopy of the priority document and the English-language translation of the priority document filed September 8, 2005, in the received date ("2005 year 9 month 5 day" v. "2004 year 1 month 26 day") and in the name of the Commissioner of the Japan Patent Office ("Ogawa Hirosi" v. "IMAI Yasuo")

Respectfully submitted,

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CERTIFICATE OF TRANSLATION

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hereby states:

(1) that I know well both the Japanese and English languages;

(2) that I translated the attached document identified as corresponding to Japanese Application No.[2002-339402] filed in Japan on [2002.11.22] from Japanese to English;

(3) that the attached English translation is a true and correct translation of Japanese Application No.[2002-339402], to the best of my knowledge and belief; and

(4) that all statements made of my own knowledge are true and that all statements made on information and belief are believed to be true and further, that these statements are made with the knowledge that willful false statements and the like are punishable by fine or imprisonment, or both, under 18 U.S.C. §1001, and that such false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: September 13, 2005 By: Noribumi Kikuchi

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ATTORNEYS AT LAW

Docket No.: 245632US0

COMMISSIONER FOR PATENTS
ALEXANDRIA, VIRGINIA 22313

RE: Application Serial No.: 10/718,573
Applicants: Toshio MORII, et al.
Filing Date: November 24, 2003
For: HIGH CONCENTRATION SILICA SLURRY
Group Art Unit: 1755
Examiner: MARCHESCHI, M. A.

SIR:

Attached hereto for filing are the following papers:
Submission Notice Regarding Priority Document(s)
Certified Copy of Priority Document JP2002-339402
Statement Regarding Priority Document and English-language Translation of Priority Document
Certified English-language Translation of Priority Document
Certificate of Translation Accuracy (1 pg., executed)

Our check in the amount of **$0.00** is attached covering any required fees. In the event any variance exists between the amount enclosed and the Patent Office charges for filing the above-noted documents, including any fees required under 37 C.F.R 1.136 for any necessary Extension of Time to make the filing of the attached documents timely, please charge or credit the difference to our Deposit Account No. 15-0030. Further, if these papers are not considered timely filed, then a petition is hereby made under 37 C.F.R. 1.136 for the necessary extension of time. A duplicate copy of this sheet is enclosed.

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SUBMISSION NOTICE REGARDING PRIORITY DOCUMENT(S)

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ALEXANDRIA, VIRGINIA 22313

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Certified copies of the Convention Application(s) corresponding to the above-captioned matter:

- ■ are submitted herewith
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- □ were submitted to the International Bureau in PCT Application Number _____
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日本国特許庁
JAPAN PATENT OFFICE

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【書類名】　明細書

【発明の名称】　高濃度シリカスラリー

【特許請求の範囲】

【請求項１】　ナトリウムおよびカリウムの不純物濃度が何れも１．０ppm以下のシリカ粉末を溶媒に分散したシリカ濃度５０wt％以上および粘度１０００mPa･s以下であることを特徴とする高濃度シリカスラリー。

【請求項２】　レーザー回折粒度分布による平均粒子径とＴＥＭ撮影による平均粒子径の比が１．３以下であって、平均一次粒子径が０．０８μm～０．８μmであるシリカ粉末を用いた請求項１に記載する高濃度シリカスラリー。

【請求項３】　シリカ濃度が５０wt％以上～８０wt％以下であることを特徴とする請求項１または２に記載する高濃度シリカスラリー。

【請求項４】　スラリー調製時の粘度と１月経過時の粘度の比が１．５以下である請求項１、２または３に記載する高濃度シリカスラリー。

【請求項５】　乾式法によって製造されたシリカ粉末を用いた請求項１～４の何れかに記載する高濃度シリカスラリー。

【請求項６】　粒子径が異なる２種類以上のシリカ粉末を用いた請求項１～５の何れかに記載する高濃度シリカスラリー。

【請求項７】　シリカ粉末と共に他の金属酸化物粉末を含む請求項１～６の何れかに記載する高濃度シリカスラリー。

【請求項８】　請求項１～７の何れか記載する高濃度シリカスラリーを用いた研磨組成物。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】

本発明は、不純物の少ない均一な粒子径のシリカ粉末を用いることによって高濃度でありながら低粘性のシリカスラリーとしたものであり、このシリカスラリーを用いた研磨性能の高い研磨組成物に関する。本発明のシリカ分散スラリーは低濃度から高濃度に至る広い濃度範囲でシリカ粉末が沈降せずに分散しており、しかも低粘性であるので、取り扱い性が良く、また経時安定性に優れ、高速研磨

や粗研磨に適する。また、高濃度で取り扱いできることから安価に供給できる利点を有する。

【０００２】

【従来の技術】

化学的研磨(CMP)において、フュームドシリカやコロイダルシリカの分散液スラリーが用いられている。シリコンウエハの研磨は通常、一次、二次、仕上げ研磨に分けられ、最後の仕上がり表面の状況はスクラッチやヘイズがないものが求められている。さらには、ウエハ表面の研磨中に金属イオン、特にナトリウムのような金属イオンが基板表層に取り込まれて金属イオン汚染を生じることがないような高純度の研磨スラリーが強く求められている。

【０００３】

【発明が解決しようとする課題】

シリカ粉末分散液について、金属汚染が少なく、研磨精度と速度に優れた研磨液を得るには、高純度のシリカ粉末を用いると共にシリカ粉末が高濃度であって分散性が良く、かつ低粘度で経時安定性に優れたスラリーであることが必要である。ところが、従来のシリカ分散スラリーのシリカ濃度は４０％程度が限界であり、シリカ濃度がこれより高いと適度な流動性を失うものが多い。また、従来の分散液はスラリーの粘度における経時安定性が低いと云う問題がある。特に微細な粒子径のシリカ粉末にこの傾向が強い。具体的には例えば平均一次粒子径７～５０nmのフュームドシリカを用いた従来のスラリーは、スラリー中でシリカ粉末が凝集体として存在する傾向が強いため、研磨時の粒子径が均一ではなく、粘度の経時変化も大きい。

【０００４】

【発明が解決しようとする課題】

本発明は、従来のシリカスラリーにおける上記問題を解決したものであり、高濃度でありながら粘性が低く、しかも粘度の経時変化が少ない高濃度シリカスラリーを提供するものである。

【０００５】

【課題を解決する手段】

すなわち、本発明は以下の構成からなる高濃度シリカスラリーとその研磨組成物が提供される。

（１）ナトリウムおよびカリウムの不純物濃度が何れも１．０ppm以下のシリカ粉末を溶媒に分散したシリカ濃度５０wt％以上および粘度１０００mPa･s以下であることを特徴とする高濃度シリカスラリー。

（２）レーザー回折粒度分布による平均粒子径とＴＥＭ撮影による平均粒子径の比が１．３以下であって、平均一次粒子径が０．０８μm～０．８μmであるシリカ粉末を用いた上記(1)に記載する高濃度シリカスラリー。

（３）　シリカ濃度が５０wt％以上～８０wt％以下であることを特徴とする上記(1)または(2)に記載する高濃度シリカスラリー。

（４）スラリー調製時の粘度と１月経過時の粘度の比が１．５以下である上記(1)、(2)または(3)に記載する高濃度シリカスラリー。

（５）乾式法によって製造されたシリカ粉末を用いた上記(1)～(4)の何れかに記載する高濃度シリカスラリー。

（６）粒子径が異なる２種類以上のシリカ粉末を用いた上記(1)～(5)の何れかに記載する高濃度シリカスラリー。

（７）シリカ粉末と共に他の金属酸化物粉末を含む上記(1)～(6)の何れかに記載する高濃度シリカスラリー。

（８）　上記(1)～(7)の何れか記載する高濃度シリカスラリーを用いた研磨組成物。

【０００６】

本発明のシリカ分散スラリーは、不純物の少ないシリカ粉末を用い、高濃度でありながら粘性が低く、しかも粘度の経時変化が少ないスラリーである。従って本発明のシリカ分散スラリーはシリコンウエハー等の研磨組成物として好適であり、ＣＭＰに用いた際に、研磨速度と研磨精度の両方の性能に優れた効果を得ることができる。

【０００７】

【発明の実施の形態】

以下、本発明を実施形態に基づいて具体的に説明する。なお、％は特に示さな

い限り重量％である。

【０００８】

本発明のスラリーは、ナトリウムおよびカリウムの不純物濃度が何れも１．０ppm以下のシリカ粉末を溶媒に分散した、シリカ濃度５０％以上であって粘度１０００mPa･s以下の高濃度シリカスラリーである。ナトリウムおよびカリウムの不純物濃度が何れも１．０ppmより高いシリカ粉末を分散させたスラリーは、これをウエハーなどの研磨に用いると、シリカ粉末に含まれているナトリウムやカリウムなどの不純物イオンが研磨中にウエハーの表層に取り込まれて汚染を生じ、研磨面にスクラッチやヘイズを生じることが多い。

【０００９】

金属汚染を防止するには、ナトリウムおよびカリウム以外の不純物についても、その不純物濃度はできるだけ少ないものが好ましい。具体的には、例えば、アルミニウム１．０ppm以下であって、硫黄、ニッケル、クロム、鉄は何れも０．５ppm以下、より好ましくは硫黄、ニッケル、クロムは何れも０．１ppm以下であるのが良い。

【００１０】

また、本発明のスラリーに用いるシリカ粉末は、レーザー回折粒度分布による平均粒子径とＴＥＭ撮影による平均粒子径の比が１．３以下であって、平均一次粒子径が０．０８μｍ～０．８μｍであるものが好ましい。シリカ粒子の平均一次粒子径が０．０８μｍより小さいものはスラリーが不安定になる傾向があり、粘度の経時変化やスラリーの沈降を生じやすい。また、平均一次粒子径が０．８０μｍより大きいシリカ粒子は、スラリー中で沈降を生じやすく、研磨に用いた場合にスクラッチ等の原因となる。

【００１１】

また、レーザー回折粒度分布による平均粒子径とＴＥＭ撮影による平均粒子径の比が１．３を超えるものは二次凝集体が多く、高濃度かつ低粘性のシリカスラリーを得るのが難しい。このように本発明のシリカ粉末は二次凝集体が少なく、二次凝集体の平均粒子径が概ね１．０μm以下のものである。さらに凝集粒子はこの１．０μm以下の範囲にその９５％以上が一山に分布しているものが好ましい。

シリカ粒子の一山の分布ピークが９５％より少なく、またはこの分布ピークにある凝集粒子の平均粒子径が１．０μｍより大きいと、スラリーが不安定であり、粘度の経時変化が大きくなる。

【００１２】

不純物濃度が少なく粒子径が上記範囲のシリカ粉末は、例えば火炎加水分解法などの乾式法で製造したフュームドシリカなどである。湿式法によって製造したシリカ粉末は金属不純物が上記濃度水準以下のものを得るのが難い。乾式法で製造したシリカ粉末としては、例えば特開２００２－３２１３に記載されている方法によって製造したものを用いるとよい。本製造方法はガス状の珪素化合物を火炎に導いて加水分解することによって非晶質シリカ微粒子を製造する方法であり、火炎温度をシリカの融点以上とし、この火炎中のシリカ濃度を０．２５kg/Nm^{3}以上に保って生成したシリカ粒子をシリカの融点以上の高温下に短時間滞留させ、平均粒径（メジアン径）０．０８～０．８０μｍおよび比表面積５～３０m^2/gの非晶質シリカ粒子を得る製造方法である。

【００１３】

上記製造方法において、原料の珪素化合物は四塩化珪素、トリクロロシラン、ジクロロシラン、メチルトリクロロシラン等のガス状で酸水素炎中に導入され、高温下で加水分解反応を生じるものが用いられる。これらの四塩化珪素等のガス状珪素化合物は蒸留精製によって原料中の不純物を容易に除去できるので高純度のシリカ粒子を製造するのに適している。

【００１４】

ナトリウムおよびカリウム等の不純物濃度が少なく、かつ粒子径が上記範囲のシリカ粉末を用いることにより、シリカ濃度５０％以上の高濃度スラリーでありながら、粘度１０００mPa·s以下、かつ粘性の経時変化が少ない低粘性シリカスラリーを得ることができる。具体的には、例えば、シリカ濃度７０％以上であって初期粘度８００mPa·s以下、スラリー調製時の粘度Ａと１月経過時の粘度Ｂの比（以下、経時粘度比と云う）が１．５以下の低粘性シリカスラリーを得ることができる。経時粘度比（Ｈ＝Ｂ／Ａ）が１．５より高いものは経時的な粘度変化が大きく、スラリーが不安定である。

【００１５】

スラリーのシリカ濃度は８０％以下が好ましい。シリカ濃度が８０％より高いと粘度が高くなり過ぎてゲル化や沈降を生じやすくなる。また、日常の温度変化や運搬･保存条件等の相違によって容易にゲル化や沈澱を生じるなど、スラリーの経時安定性が低下する。さらにスラリーの粘度が１０００mPa･sより高いと取り扱い性に問題が生じる。なお、シリカ粉末を分散する溶媒は蒸留水などの極性溶媒が望ましい。溶媒の粘度は１０mPa･s以下が適当である。

【００１６】

本発明のシリカスラリーは粒子径が異なる２種類以上のシリカ粉末を用いることができる。さらに、シリカ粉末と共に他の金属酸化物粉末、例えばアルミナ粒子、複合酸化物粒子、ドープドマテリアルなどを含むことができる。これらの金属酸化物粉末を含むことによって目的に応じた研磨が可能になる。

【００１７】

【実施例】

以下、実施例および比較例によって本発明を具体的に示す。なお、各例において粘度はＨＡＡＫＥ社のRheoStress計を用いて測定し、粒子径はＨＯＲＩＢＡ社のレーザー散乱式粒度分布計を用いて測定した。粘度は１００/ｓのせん断速度における値、経時変化粘度はスラリー調製時から１ヶ月経過後の粘度である。

【００１８】

〔実施例１〕

表１に示す不純物濃度のフュームドシリカ（商品名VP-OX30、比表面積30m^2/g、日本アエロジル社製品）を用い、このフュームドシリカを蒸留水に添加し、攪拌分散させてスラリー化し、ｐＨ８．９に調整してシリカ濃度７５％のスラリーを調製した。このスラリーの粘度と粒子径を測定した。またスラリー中の凝集粒子径および凝集一山率を測定した。この結果をスラリー化条件と共に表１に示した。

【００１９】

〔実施例２〕

表１に示す不純物濃度のフュームドシリカ（商品名VP-OX10、比表面積10m^2/g、日本アエロジル社製品）を用い、このフュームドシリカを蒸留水に添加し、攪

拌分散させてスラリー化し、ｐＨ９．２に調整してシリカ濃度８０％のスラリーを調製した。このスラリーの粘度と粒子径を測定した。またスラリー中の凝集粒子径および凝集一山率を測定した。この結果をスラリー化条件と共に表１に示した。

【００２０】

〔比較例１〕

表１に示す不純物濃度のフュームドシリカ（商品名VP-OX30、比表面積30m^2/g、日本アエロジル社製品）を用い、このフュームドシリカを蒸留水に添加し、攪拌分散させてスラリー化し、ｐＨ８．９に調整し、シリカ濃度８５％のスラリーを調製した。このスラリーの粘度と粒子径を測定した。またスラリー中の凝集粒子径および凝集一山率を測定した。この結果をスラリー化条件と共に表１に示した。

【００２１】

〔比較例２〕

蒸留水に市販のコロイダルシリカを添加し、攪拌分散させてスラリー化し、ｐＨ９．２に調整し、シリカ濃度７０％のスラリーを調製した。このスラリーの粘度と粒子径を測定した。またスラリー中の凝集粒子径および凝集一山率を測定した。この結果をスラリー化条件と共に表１に示した。

【００２２】

〔比較例３〕

比較例２において、コロイダルシリカに代えて湿式法によって製造したシリカ粉末を用いた以外は同様にしてシリカ濃度７０％のスラリーを調製した。このスラリーの粘度と粒子径を測定した。またスラリー中の凝集粒子径および凝集一山率を測定した。この結果をスラリー化条件と共に表１に示した。

【００２３】

表１に示すように、本発明に係る実施例１、２のシリカスラリーはシリカ濃度が何れも７５％以上でありながら、スラリー調製時の初期粘度は各々７００mPa·s、８００mPa·sであり、何れもスラリーの粘性が低い。さらに１月経過時のスラリー粘度は各々７３０mPa·s、８５０mPa·sであり、従って経時粘度変化が小さく安定である。一方、比較例２、３はシリカ濃度７０％においてスラリー粘度は１

０００mPa･s以上であり、低粘度スラリーを得ることができない。

【００２４】

【発明の効果】

本発明のシリカスラリーは、ナトリウムおよびカリウムの不純物濃度が何れも１．０ｐｐｍ以下のシリカ粉末を溶媒に分散させたシリカ濃度５０％以上のスラリーであり、シリカ濃度の高いにもかかわらずスラリー粘度が低く、かつ粘度の経時変化も小さい。従って、化学的研磨（ＣＭＰ）などにおいて、研磨速度が比較的速く、しかも研磨精度にも優れる。

【００２５】

【表１】

		実施例1	実施例2	比較例1	比較例2	比較例3
シリカの種類		VP-OX30	VP-OX10	VP-OX30	コロイダルシリカ	湿式シリカ
不純物濃度[ppm]	Na	<1.0	<1.0	<1.0	277	2226
	K	<1.0	<1.0	<1.0	<10	<1.0
	S	<0.1	<0.1	<0.1	<1.0	1200
	Fe	<0.5	<0.5	<0.5	<1.0	210
	Al	<1.0	<1.0	<1.0	<1.0	<1.0
	Ni	<0.1	<0.1	<0.1	<1.0	<1.0
	Cr	<0.1	<0.1	<0.1	<1.0	<1.0
スラリー	シリカ濃度%	75	80	85	70	70
	粘度(初期)[mPa･s]	700	800	>10000	>10000	>10000
	粘度(0.5ヶ月)[mPa･s]	710	830	>10000	>10000	>10000
	粘度(1ヶ月)[mPa･s]	730	850	>10000	>10000	>10000
	二次凝集粒度分布	0.13	0.39	0.16	0.51	2
	一次粒子粒度分布	0.11	0.33	0.11	0.31	特定不可
	95%以上一山	○	○	×	×	×

(注) 二次凝集と一次粒子の粒度分布はメジアン径（μm）

【書類名】　要約書

【要約】

【課題】　不純物汚染を生じることがなく、研磨速度と研磨精度に優れた高濃度のシリカスラリーを提供する。

【解決手段】　ナトリウムおよびカリウムの不純物濃度が何れも１．０ppm以下のシリカ粉末であり、好ましくは、レーザー回折粒度分布による平均粒子径とＴＥＭ撮影による平均粒子径の比が１．３以下であって平均一次粒子径が０．０８μｍ～０．８μｍのシリカ粉末を溶媒に分散したシリカ濃度５０wt％以上であって粘度１０００mPa･s以下であることを特徴とする高濃度シリカスラリー。

【選択図】　なし

特願２００２－３３９４０２

出　願　人　履　歴　情　報

識別番号　［３９００１８７４０］

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[Name of Document] Specification

[Title of the Invention] HIGH CONCENTRATION SILICA SLURRY

[What is claimed is]

[Claim 1] A high concentration silica slurry having a silica concentration of 50 wt. % or more and a viscosity of 1000 mPa·s or less, wherein a silica powder is dispersed in an solvent, and impurity concentrations of sodium and potassium are 1.0 ppm or less in said silica powder.

[Claim 2] The high concentration silica slurry according to claim 1, wherein said silica powder has a ratio between an average particle diameter measured by a laser diffraction method and that measured by a TEM photography observation method being 1.3 or less, and has an average primary particle diameter of from 0.08 μm to 0.8 μm

[Claim 3] The high concentration silica slurry according to claim 1 or 2,

wherein the silica concentration is from 50 wt. % or more to 80 wt. % or less.

[Claim 4] The high concentration silica slurry according to claim 1, 2 or 3,

wherein ratio between a viscosity of a slurry after one month and that at the time of preparing is 1.5 or less.

[Claim 5] The high concentration silica slurry according to any one of claims 1 to 4,

wherein the silica powder produced by a dry process is used.

[Claim 6] The high concentration silica slurry according to any one of

claims 1 to 5,

wherein 2 or more kinds of silica powders having different particle diameters are used.

[Claim 7] The high concentration silica slurry according to any one of claims 1 to 6,

wherein a metal oxide powder is also included together with the silica powder.

[Claim 8] A polishing composite,

wherein the high concentration silica slurry according to any one of claims 1 to 7 is used.

[Background of the Invention]

[Field of the invention]

[0001]

The present invention relates to a silica slurry having low viscosity even when having a high silica concentration by using a silica powder having a uniform particle diameter and low agglomeration, and also relates to a polishing composite having excellent polishing performance using the silica slurry. As for the silica dispersion slurry of the present invention, the silica powder is dispersed in the wide concentration range from low to high without precipitating, and the viscosity is low. Thus, this slurry has excellent handling, is stable with the passage of time, and suitable for high-speed polishing or rough polishing. Further, since the silica dispersion slurry can be handled with the high concentration, the supplying cost of the slurry is low.

[Description of the Prior Art]

[0002]

In a chemical mechanical polishing (CMP), a dispersion slurry of fumed silica or colloidal silica has been used. A general polishing process of a silicon wafer is classified with 3 processes, i.e., a primary polishing, a secondary polishing and a finishing polishing, and it is required in the finishing polishing that the final condition of the surface has no scratch and haze. Furthermore, the high purity polishing slurry is strongly required in order to prevent metal ionic contaminations aroused by metal ions, such as sodium especially, into a surface layer of a substrate at the time of polishing

the wafer surface.

[Problems to be solved]

[0003]

As for the dispersion liquid of the silica powder, in order to obtain a polishing liquid having few metal contaminations and excellent polishing accuracy and speed, it is necessary that the slurry has the high concentration silica powder, excellent dispersibility, a low viscosity, and excellent stability with the passage of time, in addition using the high purity silica powder. However, the silica concentration of the conventional silica dispersion slurry is limited about 40 %. If the silica concentration is more than 40 %, the suitable flowability is lost in many cases. Further, there is a problem that the conventional dispersion liquid has the low stability with the passage of time in the viscosity of the slurry. Especially, in the case of the silica powder having the fine particle diameter, this problem is large. More particularly, for example, as for the conventional slurry using the fumed silica having the average primary particle diameter of from 7 to 50 nm, since the silica powder exists as agglomerates in the slurry in many cases, the particle diameter at the time of polishing is not uniform, and the viscosity change with the passage of time is large.

[0004]

[Problems to be solved]

The present invention solves the above-mentioned problems in the conventional silica slurry, and provides the high concentration silica slurry

having low viscosity and little viscosity change with the passage of time even when having high concentration.

[0005]

[Means for Solving Problem]

That is, the present invention provides the high concentration silica slurry and the polishing composite comprising the following constitutions.

(1) A high concentration silica slurry having a silica concentration of 50 wt. % or more and a viscosity of 1000 mPa·s or less,

wherein a silica powder is dispersed in an solvent, and impurity concentrations of sodium and potassium are 1.0 ppm or less in the silica powder.

(2) The high concentration silica slurry according to above (1),

wherein the silica powder has a ratio between an average particle diameter measured by a laser diffraction method and that measured by a TEM photography observation method being 1.3 or less, and has an average primary particle diameter of from 0.08 μm to 0.8 μm.

(3) The high concentration silica slurry according to above (1) or (2),

wherein the silica concentration is from 50 wt. % or more to 80 wt. % or less.

(4) The high concentration silica slurry according to above (1), (2) or (3),

wherein a ratio between the viscosity of the slurry after one month and that at the time of preparing is 1.5 or less.

(5) The high concentration silica slurry according to any one of above (1) to (4),

wherein the silica powder produced by a dry process is used.

(6) The high concentration silica slurry according to any one of above (1) to (5),

wherein 2 or more kinds of silica powders having different particle diameters are used.

(7) The high concentration silica slurry according to any one of above (1) to (6),

wherein a metal oxide powder is also included together with the silica powder.

(8) A polishing composite,

wherein the high concentration silica slurry according to any one of above (1) to (7) is used.

[0006]

The silica dispersion slurry of the present invention uses the silica powder having the few impurities and has the low viscosity and the little viscosity change with the passage of time even when having the high concentration. Therefore, the silica dispersion slurry of the present invention is suitable as the polishing composite of a silicon wafer or the like. In addition, when the silica dispersion slurry is used in CMP, it is possible to obtain the excellent effect in both performances of the polishing speed and accuracy.

[0007]

[Detailed description of the preferred embodiment]

Hereinafter, the present invention is described more concretely on the basis of the preferred embodiment. In addition, % is the weight % unless it is shown especially.

[0008]

The slurry of the present invention is the high concentration silica slurry having the silica concentration of 50 % or more and the viscosity of 1000 mPa·s or less, wherein the silica powder is dispersed in the solvent and the impurity concentrations of sodium and potassium are 1.0 ppm or less in the silica powder. When the slurry dispersed with the silica powder having each impurity concentration of sodium and potassium of more than 1.0 ppm is used in polishing the wafer or the like, ionic impurity of sodium or potassium contained in the silica powder is taken into to thereby arise the contaminations, so that the scratch or the haze occurs on the polished surface in many cases.

[0009]

In order to prevent the metal contaminations, it is preferable that the concentrations of the impurities other than sodium and potassium are kept as low as possible. More particularly, for example, it is preferable that aluminum is less than 1.0 ppm, and each of sulfur, nickel, chromium and iron is less than 0.5 ppm. More preferably, each of sulfur, nickel and chromium is less than 0.1 ppm.

[0010]

Further, it is preferable that the silica powder used in the slurry of the present invention has the ratio between an average particle diameter measured by a laser diffraction method and that measured by a TEM photography observation method being 1.3 or less, and has the average primary particle diameter of from 0.08 μm to 0.8 μm. When the silica powder having the average primary particle diameter of smaller than 0.08 μ

m is used, the slurry becomes unstable in many cases, so that the viscosity change with the passage of time and the precipitation in the slurry occur easily. On the other hand, when the silica powder having the average primary particle diameter of larger than 0.80 μm is used, the powder is precipitated easily in the slurry, and causes the scratch or the like at the time of polishing.

[0011]

Further, as for the silica powder having the ratio between an average particle diameter measured by a laser diffraction method and that measured by a TEM photography observation method being 1.3 or more, there are many secondary agglomerates, so that the silica slurry having the high concentration and the low viscosity is hardly obtained. Thus, as for the silica powder of the present invention, there are few secondary agglomerates, and the average particle diameter of the secondary agglomerates is about 1.0 μm or less. Further, as for the agglomerated particles, it is preferable that the agglomerated particles in one peak of a size distribution curve are more than 95 % where the one peak is in the range of the particle diameter of less than 1.0 μm. When the agglomerated particles in one peak of the size distribution curve are less than 95 % or the average particle diameter of the agglomerates in this peak is more than 1.0 μm, the slurry becomes unstable, and the viscosity change with the passage of time becomes large.

[0012]

The silica powder having the low impurity concentration and the particle diameter within the above range is, for example, the fumed silica or the like produced by the dry process, such as a flame hydrolysis method or

the like. As for the silica powder made by a wet process, it is difficult to obtain the slurry having the impurity concentration of less than the above-mentioned level. As the silica powder made by the dry process, for example, it is preferable to use the silica powder made by the method described in Japanese Patent Application Laid Open No. 2002-3213. This production method is to produce an amorphous fine silica powder by introducing a gaseous silicon compound into a flame and hydrolyzing it. In this production method, the amorphous silica powder having the average particle diameter (median diameter) of from 0.08 to 0.8 μm and the specific surface area of from 5 to 30 m^2 / g, is produced by making the flame temperature to have more than a melting point of silica, growing the silica particle while keeping the silica concentration in the flame of 0.25 kg/Nm^3 or more, and staying the grown silica particle at the high temperature of more than the melting point in a short time.

[0013]

In the production method, as a raw material, a silicon compound, such as, silicon tetrachloride, trichlorosilane, dichlorosilane, methyltrichlorosilane or the like is used and carried out the hydrolysis reaction at the high temperature by introducing into the oxy-hydrogen flame in gaseous state. These gaseous silicone compounds, such as the silicon tetrachloride or the like, are suitable to produce the high purity silica particle since the impurity in the raw material can be removed easily by a distillation refining.

[0014]

By using the silica particle having the low impurity concentrations of

sodium and potassium and the particle diameter within the above range, it is possible to obtain the low viscosity silica slurry having the viscosity of 1000 mPa·s or less and the little viscosity change with the passage of time, even when having the high silica concentration of more than 50 %. More particularly, for example, it is possible to obtain the low viscosity silica slurry, in which the silica concentration is 70 % or more, the initial viscosity is 800 mPa·s. or less, and the ratio between the viscosity B after one month and the viscosity A at the time of preparing (hereinafter, it is said to as the viscosity ratio with the passage of time) is 1.5 or less. When the viscosity ratio with the passage of time (H=B/A) is more than 1.5, the viscosity change with the passage of time is large and the slurry is unstable.

[0015]

It is preferable that the silica concentration of the slurry is 80 % or less. When the silica concentration is more than 80 %, the viscosity becomes too high to thereby generate gelation or precipitation easily. Further, the stability with the passage of time of the slurry is decreased, by which the gelation or the precipitation is generated easily by the daily temperature change or the difference of transportation or preservation conditions. Furthermore, when the viscosity of the slurry is more than 1000 mPa·s, there is a problem of a handling property. In addition, as a solvent dispersing the silica powder, a polar solvent, such as the distilled water or the like, is desirable. It is suitable that the viscosity of the solvent is 10 mPa·s or less.

[0016]

The silica slurry of the present invention can use 2 or more kinds of the silica powders having the different particle diameters. Further, other

metal oxide powders, such as an alumina powder, composite oxide powders, doped materials or the like, can be contained together with the silica powder. By containing these metal oxide powders, it becomes possible to polish according to the purpose.

[0017]

[Example]

Hereinafter, the present invention is described more concretely with examples and comparison examples. In addition, in each example, the viscosity is measured using Rheo Stress meter produced by HAAKE Co., and the particle diameter is measured using a laser dispersion type particle size distribution meter produced by HORIBA Co. The viscosity is the value at the shearing speed of 100 / s, and the viscosity change with the passage of time is measured after one month from the time of preparing the slurry.

[0018]

[Example1]

The slurry having the silica concentration of 75 % was prepared by steps of using the fumed silica produced by Nippon Aerosil Co., Ltd. and having the impurity concentration shown in Table 1 (a trade name was VP－OX30 and the specific surface area was 30 m^2/g), adding the fumed silica to the distilled water, stirring and dispersing it to thereby make the slurry, and adjusting pH to have 8.9. The viscosity and the particle diameter of the slurry were measured. Further, the agglomerated particle diameter in the slurry and the agglomeration ratio in one peak of the size distribution curve were measured. These results were shown in Table 1 with the making slurry conditions.

[0019]

[Example 2]

The slurry having the silica concentration of 80 % was prepared by steps of using the fumed silica being produced by Nippon Aerosil Co., Ltd. and having the impurity concentration shown in Table 1 (the trade name was VP－OX10 and the specific surface area was 10 m^2/g), adding the fumed silica to the distilled water, stirring and dispersing it to thereby make the slurry, and adjusting pH to have 9.2. The viscosity and the particle diameter of the slurry were measured. Further, the agglomerated particle diameter in the slurry and the agglomeration ratio in one peak of the size distribution curve were measured. These results were shown in Table 1 with the making slurry conditions.

[0020]

[Comparison example 1]

The slurry having the silica concentration of 85 % was prepared by steps of using fumed silica being produced by Nippon Aerosil Co., Ltd and having the impurity concentration shown in Table 1 (a trade name was VP－OX30 and the specific surface area was 30 m^2/g), adding the fumed silica to the distilled water, stirring and dispersing it to thereby make the slurry, and adjusting pH to have 8.9. The viscosity and the particle diameter of the slurry were measured. Further, the agglomerated particle diameter in the slurry and the agglomeration ratio in one peak of the size distribution curve were measured. These results were shown in Table 1 with the making slurry conditions.

[0021]

[Comparison example 2]

The slurry having the silica concentration of 70 % was prepared by steps of adding commercial colloidal silica to the distilled water, stirring and dispersing it to thereby make the slurry, and adjusting pH to have 9.2. The viscosity and the particle diameter of the slurry were measured. Further, the agglomerated particle diameter in the slurry and the agglomeration ratio in one peak of the size distribution curve were measured. These results were shown in Table 1 with the making slurry conditions.

[0022]

[Comparison example 3]

The slurry having the silica concentration of 70 % was prepared by the same process in the comparison example 2 except using the silica powder produced by the wet process instead of the colloidal silica. The viscosity and the particle diameter of the slurry were measured. Further, the agglomerated particle diameter in the slurry and the agglomeration ratio in one peak of the size distribution curve were measured. These results were shown in Table 1 with the making slurry conditions.

[0023]

As shown in Table 1, in the case of each silica slurry of examples 1 and 2 of the present invention, each initial viscosity of the slurry at the time of preparing was 700 mPa·s and 800 mPa·s respectively, even when each silica concentration was more than 75 %. So, the each viscosity of the silica slurries was low. Further, each viscosity of the silica slurries after one month was 730 mPa·s and 850 mPa·s respectively, so that each slurry had little viscosity change with the passage of time and was stable. On the other hand,

in the case of comparison examples 1 and 2, when the silica concentration was 70 %, the viscosity of the slurry was more than 10000 mPa·s, so that the low viscosity slurry could not be obtained.

[0024]

[Effectiveness of the Invention]

The silica slurry of the present invention is the slurry having the silica concentration of 50 % or more wherein the silica powder is dispersed in the solvent and the impurity concentrations of sodium and potassium in the silica powder are 1.0 ppm or less respectively. Further, this silica slurry has a low viscosity and little viscosity change with the passage of time even when having high concentration. Therefore, in the chemical mechanical polishing (CMP), the polishing speed is comparatively high, and the polishing accuracy is excellent

[0025]

[Table 1]

		Example 1	Example 2	Comparison example 1	Comparison example 2	Comparison example 3
Kinds of Silica		VP-OX30	VP-OX10	VP-OX30	Colloidal Silica	Silica by Wet Process
Impurities Concentration (ppm)	Na	< 1.0	< 1.0	< 1.0	277	2226
	K	< 1.0	< 1.0	< 1.0	< 10	< 10
	S	< 0.1	< 0.1	< 0.1	< 1.0	1200
	Fe	< 0.5	< 0.5	< 0.5	< 1.0	210
	Al	< 1.0	< 1.0	< 1.0	< 1.0	< 1.0
	Ni	< 0.1	< 0.1	< 0.1	< 1.0	< 1.0
	Cr	< 0.1	< 0.1	< 0.1	< 1.0	< 1.0
Slurry	Silica Concentration %	75	80	85	70	70
	Viscosity (Initial) [mPa·s]	700	800	> 10000	> 10000	> 10000
	Viscosity (After 0.5 month) [mPa·s]	710	830	> 10000	> 10000	> 10000
	Viscosity (After 1 month) [mPa·s]	730	850	> 10000	> 10000	> 10000
	Secondary Agglomeration Particle Size Distribution	0.13	0.39	0.16	0.51	2
	Primary Particle Particle Size Distribution	0.11	0.33	0.11	0.31	Could not be measured
	One Peak of 95 % or more	○	○	×	×	×

(Note) Particle size distributions of Secondary Agglomeration and Primary Particle are a median diameter (μ m)

Industrial Applicability

The silica dispersion slurry of the present invention uses a silica powder having few impurities and has low viscosity and a little viscosity change with the passage of time even when having a high concentration. Therefore, the silica dispersion slurry of the present invention is suitable as a polishing composite of a silicon wafer or the like. In addition, when the silica dispersion slurry is used in CMP, it is possible to obtain the excellent effect in both of a polishing speed and accuracy.

[Name of Document] Abstract

[Summary]

[PROBLEM TO BE SOLVED]: To provide a high concentration silica slurry excellent in polishing speed and accuracy, without impurity contaminations.

[SOLUTION]: A high concentration silica slurry having a silica concentration of 50 wt.% or more and a viscosity of 1000 mPa·s or less, wherein a silica powder is dispersed in a solvent and each impurity concentration of sodium and potassium is 1.0 ppm or less respectively. An average primary particle diameter of the silica is 0.08 μm to 0.8 μm and a ratio between an average particle diameter measured by laser diffraction particle size distribution method and that measured by a TEM observation is 1.3 or less.

Applicant history information

Identification Number : [390018740]

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[Reason of Conversion] : Change in residence of applicant

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